B.Sc. I-Semester (NEP)

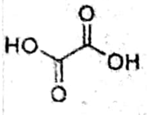
Organic Chemistry Practical

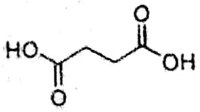
Department of Chemistry
A.M.U., Aligarh
Session: 2022-2023

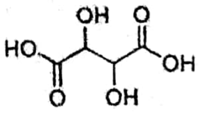
OBJECT: Identification of the following organic compounds.

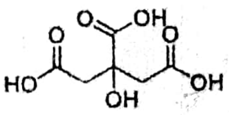
1. *Carboxylic acids*

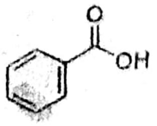
(a) Aliphatic compounds: Oxalic acid, Succinic acid, Tartaric acid, and Citric acid.
(b) Aromatic compounds: Benzoic acid, and Salicylic acid.

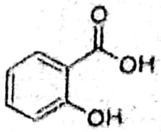

Oxalic acid


Succinic acid


Tartaric acid

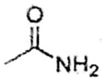

Citric acid

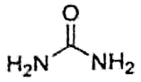

Benzoic acid

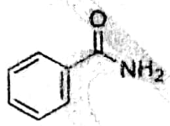

Salicylic acid

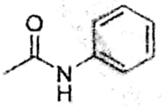
2. *Amides/Anilide*

(a) Aliphatic compounds: Acetamide, and Urea.
(b) Aromatic compounds: Benzamide, and Acetanilide.


Acetamide


Urea


Benzamide


Acetanilide

A SYSTEMATIC SCHEME FOR THE IDENTIFICATION OF THE ORGANIC COMPOUND:

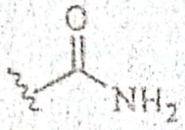
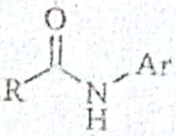
I. Preliminary Test: *Flame test/Ignition test*
II. Detection of Functional Group: *Functional group test*
III. Detection of compounds: *Confirmatory Test*

I. Preliminary test

Experiment	*Observation*	*Inference*
Flame test	Sooty flame	Maybe an aromatic compound or aliphatic compound containing a small proportion of hydrogen e.g. $CHCl_3$, CCl_4
	Non-sooty flame	Aliphatic compound
	Substances with char sound	Carbohydrate
	Smell of ammonia	Nitrogenous compounds like Urea, amides, etc.

II. Functional group test

Functional group	*Experiment*	*Observation*	*Inference*
Carboxylic acid –COOH	**Litmus Test:** Add a drop of a given aqueous solution of an organic compound on blue litmus paper.	Blue litmus changes to red.	Maybe –COOH group present.
	$R\text{-}COOH + H_2O \rightarrow R\text{-}COO^- + H_3O^+$		
	Sodium bicarbonate test ($NaHCO_3$): Dissolve the given	Evolution of brisk effervescence and colorless gas gives a	–COOH group is confirmed.

	organic compound in water and add $NaHCO_3$ solution or solid to it.	white precipitate with limewater.	
Amide 	**NaOH test:** 0.2 gm or 0.5 mL aqueous solution of compound + 1 mL of dil. NaOH solution + Heat. Then, add excess dil. NaOH solution in the mixture + Boil. (a) Keep HCl-dipped paper or glass rod on the mouth of the test tube. (b) Keep the moist red litmus on the mouth of the test tube.	Smell of ammonia is produced. Evolution of ammonia gas. White fumes of ammonium chloride (NH_4Cl) are produced. Red litmus turns blue	Indicate amide group is present. Indicate amide group is present.
Anilide 	**Tafel's test:** Aqueous solution of compound add 5 mL of conc. H_2SO_4 and Shake well. Then, add 0.2 gm of $K_2Cr_2O_7$.	Red to violet color is obtained. Stand for a few minutes, color changes to green.	Indicates anilide (ArNHCOR) is present.
	Hydrolysis Test (Azo dye test): In an aqueous solution of compound add 5 mL of dil. HCl. Boil the mixture and cool. Then, add 2 mL of $NaNO_2$ solution and cool again. And add 0.5 mL alkaline β-naphthol solution.	An orange dye is obtained.	Indicates anilide (ArNHCOR) is present.

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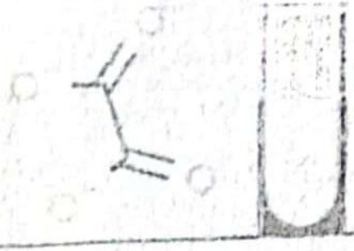
III. Confirmatory test

Carboxylic acids			
Compounds	*Experiment*	*Observation*	*Inference*
1. Oxalic acid Crystalline solid, white, odorless, soluble in cold water	**$KMnO_4$ test:** To 3 mL aqueous solution of compound, add 1 mL dil. H_2SO_4 and 4-5 drops of $KMnO_4$ and then warm on flame.	Purple colour of $KMnO_4$ is decolorized.	Oxalic acid may be present.
	$CaCl_2$ test: To 3 mL neutral solution* of given compound, add 2 mL $CaCl_2$ solution.	Immediate white ppt (in cold) of calcium oxalate are obtained. 	Oxalic acid is confirmed.

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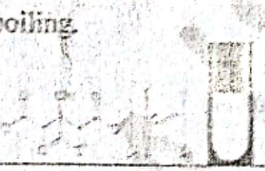
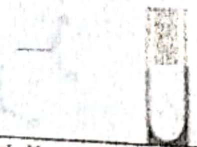
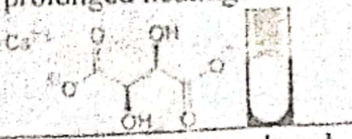
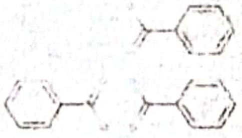
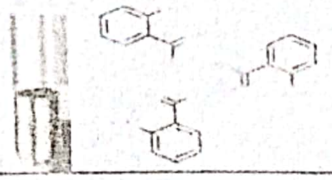
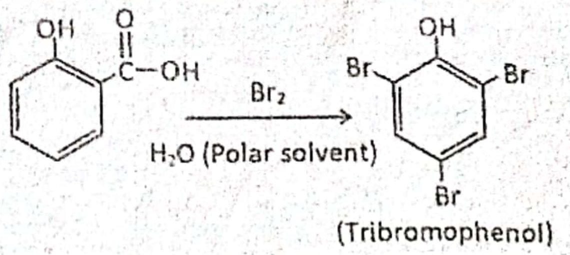
	Blue ring test: To an aqueous solution of a given compound, add Resorcinol (2-3 flakes). Heat and then cool the mixture. Add a few mL of conc. H_2SO_4 along the sides of the test tube.	Appearance of the blue ring at the junction of two layers.	Oxalic acid is confirmed.
Preparation of neutral solution*: **Method I:** Aqueous solution of compound + few drops of NH_4OH + Heat (till no smell of ammonia) + Cool. **Method II:** Aqueous solution of compound + few drops of dil. NaOH solution.			
2. Citric acid Crystalline solid, white, odorless.	**$CaCl_2$ test:** To 3 mL neutral solution* of given compound, add 2 mL $CaCl_2$ solution.	White ppt of calcium citrate is obtained after boiling. 	Citric acid is confirmed.
	Sodium nitroprusside test: To an aqueous solution of compound, add ½ mL conc. H_2SO_4 and Heat. Then, cool the resulting yellow solution and dilute it with H_2O and add NaOH solution (to make it alkaline). And add a few drops of freshly prepared sodium nitroprusside. Sodium nitroprusside dihydrate	Solution turns yellow (CO & CO_2 gas evolved). An intense red color appeared.	Citric acid is confirmed.
3. Succinic acid Crystalline solid, white, odorless.	**$CaCl_2$ test:** To 3 mL neutral solution* of given compound, add 2 mL $CaCl_2$ solution and boil.	White ppt of calcium succinate are obtained after scratching the sides of the test tube with a glass rod. 	Succinic acid is confirmed.
	Fluorescein test: Take a few crystals of succinic acid, 1-2 crystals of resorcinol, and ½ mL of conc. H_2SO_4 in a dry test tube. Heat the mixture for a few minutes, cool, and then pour the mixture into a beaker containing 100 mL of H_2O and add dil. NaOH (excess).	Initially, the red color appeared which shows green fluorescence (green fluorescence appears well after dilution with water).	Succinic acid is confirmed.

4. Tartaric acid Crystalline solid, white, odorless.	**$CaCl_2$ test:** To 3 mL neutral solution* of given compound, add 2 mL $CaCl_2$ solution and boil.	White ppt of calcium tartrate are obtained after prolonged heating. 	Tartaric acid is confirmed.
	Tollen's test: To a neutral solution* of a given compound, add Tollen's reagent and then warm the mixture on a water bath.	Silver mirror is produced. 	Tartaric acid is confirmed.
	$AgNO_3 + NH_4OH \longrightarrow \underset{\text{Tollen's reagent}}{[Ag(NH_3)_2]^+ \cdot {}^-OH} \longrightarrow 2AgOH \longrightarrow \underset{\text{Simplest reacting species}}{Ag_2O}$ $RCHO + Ag_2O \longrightarrow RCOOH + Ag\downarrow$		
	H_2SO_4 test: Heat a little amount of tartaric acid with conc. H_2SO_4 on a flame with shaking.	Charring (blackening) is observed with the evolution of CO, CO_2, and SO_2.	Tartaric acid is confirmed.
5. Benzoic acid	**$FeCl_3$ test:** To a neutral solution of a given compound add neutral $FeCl_3$ solution.	Buff-colored ppt is obtained. 	Benzoic acid is confirmed.
	$3C_6H_5COOH + FeCl_3 \longrightarrow [C_6H_5COO^-]_3\,[Fe^{3+}] + 3HCl$ *Ferric benzoate* *(Buff colored ppt)*		
6. Salicylic acid	**$FeCl_3$ test:** To a neutral solution of a given compound add neutral $FeCl_3$ solution.	Violet or purple-colored ppt (Ferric Salicylate) is obtained. 	Salicylic acid is confirmed.
	$3C_6H_5{\cdot}OH{\cdot}COOH + FeCl_3 \longrightarrow [C_6H_5{\cdot}OH{\cdot}COO^-]_3\,[Fe^{3+}] + 3HCl$ *Ferric salicylate*		
	Br_2 test: To an aqueous solution of a given compound add a few drops of Br_2 water.	White or yellow colored ppt is obtained.	Salicylic acid is confirmed.
	 OH O C—OH Br₂ H₂O (Polar solvent) OH Br Br Br (Tribromophenol)		
	Oil of wintergreen test: Take a compound in a test tube, add CH_3OH and conc. H_2SO_4 and heat it.	Smell of oil of wintergreen (like an iodex smell).	Salicylic acid is confirmed.

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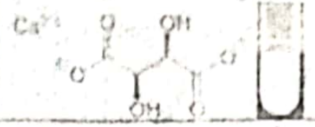
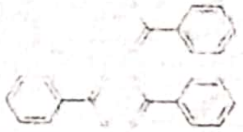
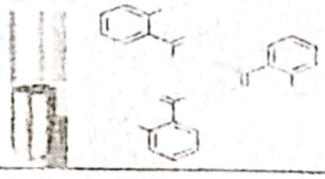
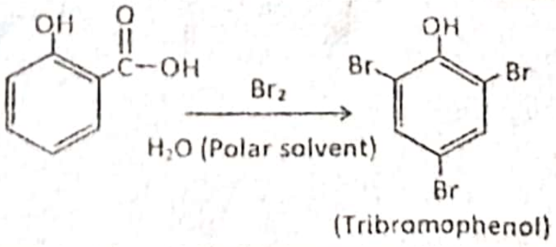
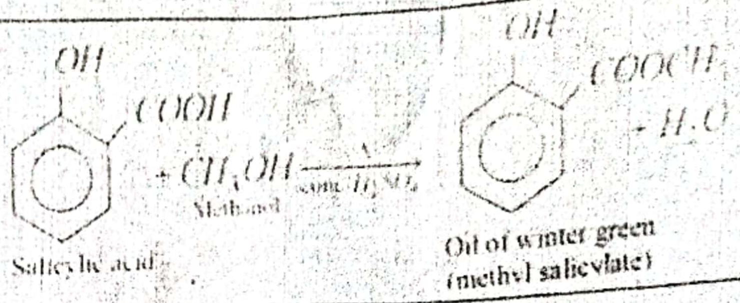
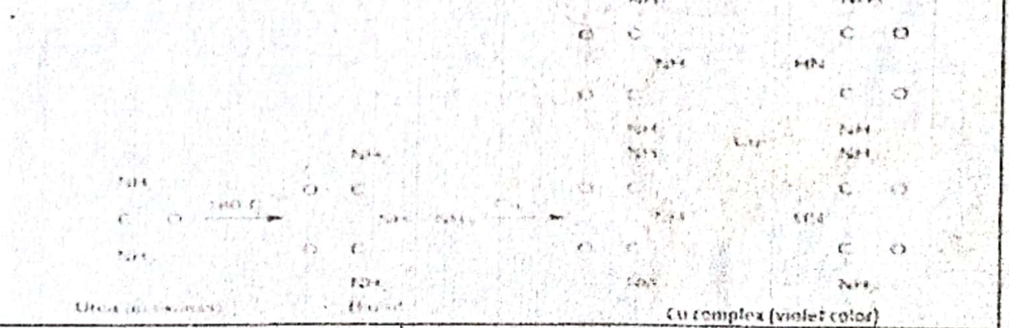
4. Tartaric acid Crystalline solid, white, odorless.	**$CaCl_2$ test:** To 3 mL neutral solution* of given compound, add 2 mL $CaCl_2$ solution and boil.	White ppt of calcium tartrate are obtained after prolonged heating.  	Tartaric acid is confirmed.
	Tollen's test: To a neutral solution* of a given compound, add Tollen's reagent and then warm the mixture on a water bath.	Silver mirror is produced. 	Tartaric acid is confirmed.
	$AgNO_3 + NH_4OH \longrightarrow [Ag(NH_3)_2]^+ \cdot {}^-OH \longrightarrow 2AgOH \longrightarrow Ag_2O$ *Tollen's reagent* — *Simplest reacting species* $RCHO + Ag_2O \longrightarrow RCOOH + Ag\downarrow$		
	H_2SO_4 test: Heat a little amount of tartaric acid with conc. H_2SO_4 on a flame with shaking.	Charring (blackening) is observed with the evolution of CO, CO_2, and SO_2.	Tartaric acid is confirmed.
5. Benzoic acid	**$FeCl_3$ test:** To a neutral solution of a given compound add neutral $FeCl_3$ solution.	Buff-colored ppt is obtained. 	Benzoic acid is confirmed.
	$3C_6H_5COOH + FeCl_3 \longrightarrow [C_6H_5COO^-]_3\,[Fe^{3+}] + 3HCl$ *Ferric benzoate* *(Buff colored ppt)* 		
6. Salicylic acid	**$FeCl_3$ test:** To a neutral solution of a given compound add neutral $FeCl_3$ solution.	Violet or purple-colored ppt (Ferric Salicylate) is obtained. 	Salicylic acid is confirmed.
	$3C_6H_5{\cdot}OH{\cdot}COOH + FeCl_3 \longrightarrow [C_6H_5{\cdot}OH{\cdot}COO^-]_3\,[Fe^{3+}] + 3HCl$ *Ferric salicylate*		
	Br_2 test: To an aqueous solution of a given compound add a few drops of Br_2 water.	White or yellow colored ppt is obtained.	Salicylic acid is confirmed.
	 OH O C—OH Br₂ H₂O (Polar solvent) OH Br Br Br (Tribromophenol)		
	Oil of wintergreen test: Take a compound in a test tube, add CH_3OH and conc. H_2SO_4 and heat it.	Smell of oil of wintergreen (like an iodex smell).	Salicylic acid is confirmed.

	 OH COOH + CH₃OH Methanol conc. H₂SO₄ Salicylic acid OH COOCH₃ + H₂O Oil of winter green (methyl salicylate)		
Amides/Anilide			
1. Acetamide White, crystalline solid, soluble in water	**NaOH test:** Take a compound and boil it with dil. NaOH.	Ammonia gas is evolved.	Acetamide may be present.
	H_2SO_4 test: Take a compound and boil it with dil. H_2SO_4.	Vinegar smell is obtained (due to the formation of acetic acid).	Acetamide is confirmed.
2. Urea White, crystalline solid, soluble in water	**NaOH test:** Take a compound and boil it with dil. NaOH.	Ammonia gas is evolved.	Urea may be present.
	Biuretic test: Take a compound in a dry test tube and Heat it (until it completely melts and all ammonia has evolved).	A crystalline biuret is formed (due to the solidification of liquid).	
	Dissolve the solid residue (crystalline biuret) in a few mL of H_2O and dil. NaOH solution then add $CuSO_4$ solution dropwise in a mixture.	A purple (violet) color is obtained.	Urea is confirmed.
			
3. Benzamide White, crystalline solid, soluble in hot water	**Benzoic acid test:** Boil 0.5 gm of a compound with 10 mL of dil. NaOH and then cool. Next, acidify the solution with dil. H_2SO_4	White ppt of benzoic acid is obtained (shiny crystals).	Benzamide is confirmed.
	Bitter almond test: Heat a little amount of the compound with sodalime.	Smell of bitter almond is observed (due to the formation of benzaldehyde and benzaldehyde also called oil of bitter almonds).	Benzamide is confirmed.
4. Acetanilide	**$K_2Cr_2O_7$ Test:** Heat the compound with a pinch of $K_2Cr_2O_7$ crystals and add 2-3 drops of conc. H_2SO_4.	The red color changing to green is observed.	Acetanilide is confirmed.